USEPA REGION 9 LABORATORY
RICHMOND, CALIFORNIA

STANDARD OPERATING PROCEDURE 375

LOW LEVEL SEMIVOLATILE ORGANICS ANALYSIS

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1 SCOPE AND APPLICABILITY

This Standard Operating Procedure (SOP) describes the procedures used for the low level analysis of selected semivolatile organic compounds by gas chromatography/mass spectrometry (GC/MS) with selected ion monitoring (SIM) in extracts prepared from solid or liquid samples by EPA SW-846 extraction and cleanup methods appropriate to the sample matrix. This SOP is based on procedures contained in EPA Method SW 846 8270D. Deviations from Method 8270 are described in Appendix A. Analytes and Quantitation Limits (QLs) for this analysis are listed in Appendix B.

Sample extracts are prepared using EPA Region 9 Lab SOP 290 *Extraction of Soil Samples Using Pressurized Fluid* or SOP 265 *Extraction of Low Level Phenols and Polycyclic Aromatic Hydrocarbons from Water*.

The applicability of this procedure to specific project data quality objectives must be assessed on a case-by-case basis. The quality control (QC) criteria specified in this procedure do not meet compliance criteria for either drinking water or NPDES monitoring projects.

2 METHOD SUMMARY

Sample extracts are injected into a gas chromatograph (GC) with a mass spectrometer (MS) detector. Analytes are separated in a narrow bore fused silica capillary GC column in a temperature-controlled oven and detected by the MS in selected ion monitoring (SIM) mode. Each target and surrogate analyte is quantitated using the average response factors from the most recent initial calibration.

Target analytes of interest are identified in the sample extract by comparing the selected characteristic ion(s) and GC retention time of the analyte to the characteristic ion(s) and retention time of standards analyzed under the same conditions.

3 DEFINITIONS

A list of terms and definitions specific to this procedure appears below. For terms and acronyms in general use at the EPA Region 9 Laboratory refer to Appendix A of the Laboratory Quality Assurance Plan.

There are no terms or definitions specific to this procedure.

4 SAFETY AND HEALTH

All laboratory operations must follow health and safety requirements outlined in current versions of the EPA Region 9 Laboratory Chemical Hygiene Plan and the Region 9 Laboratory Business Plan. Potential hazards specific to this SOP as well as pollution prevention and waste management requirements are described in the following sections.

4.1 Chemical Hazards

Due to the unknown and potentially hazardous characteristics of samples, all sample handling and preparation should be performed in a well-vented laboratory fume hood.

The toxicity and carcinogenicity of each reagent used in this method may not be fully established. Each chemical should be regarded as a potential health hazard and exposure to them should be minimized by good laboratory practices. Refer to the Material Safety Data Sheets located in Room 118 (library) and the LAN at I:\MSDS IMAGES for additional information.

Dichloromethane is a suspected carcinogen. Effects of overexposure: acute inhalation or ingestion causes mild central nervous system depression. The primary toxic effect is narcosis. Other toxic effects are pulmonary edema, encephalopathy, and hemolysis. Dichloromethane irritates the eyes, skin, and respiratory tract. No systemic effects have been reported in humans, although excessive concentrations have caused cancer and liver and kidney damage in animals.

Emergency and first aid:

- Inhalation: immediately remove to fresh air. If not breathing, administer mouth to mouth rescue breathing. If there is no pulse, administer cardiopulmonary resuscitation (CPR), contact physician immediately.
- Eye contact: rinse with copious amounts of water for at least 15 minutes. Get emergency medical assistance.
- Skin contact: flush thoroughly for at least 15 minutes. Wash affected skin with soap and water. Remove contaminated clothing and shoes. Wash clothing before re use, and discard contaminated shoes. Get emergency medical assistance.
- Ingestion: call local poison control center for assistance. Contact physician immediately. Never induce vomiting or give anything by mouth to a victim unconscious or having convulsions.

Some method analytes have been tentatively classified as known or suspected human or mammalian carcinogens. Stock standard solutions of these compounds must be prepared in a fume hood. Routine procedures in this SOP do not require contact with concentrated solutions or neat materials. All standard preparation procedures associated

with this SOP should be performed in a fume hood wearing protective clothing (lab coats) and safety glasses.

4.2 Equipment and Instruments

Follow the manufacturer's safety instructions whenever performing maintenance or troubleshooting work on equipment or instruments. Unplug the power supply before working on internal instrument components. Use of personal protective equipment may be warranted if physical or chemical hazards are present.

4.3 Pollution Prevention

Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operations. The EPA Region 9 Laboratory places pollution prevention as the management option of first choice with regard to environmental management. Whenever feasible, laboratory personnel shall use pollution prevention techniques to address waste generation. When wastes cannot be feasibly reduced, recycling is the next best option. The *EPA Region 9 Laboratory Environmental Management System* provides details regarding efforts to minimize waste.

Minimize waste through the judicious selection of volumes for reagents and standards to prevent the generation of waste due to expiration of excess materials. Reduce the volume of any reagent or standard described in Sections 7.2 or 7.3 so long as good laboratory practices are adhered to regarding the accuracy and precision of the glassware, syringes, and/or analytical balances used to prepare the solution. Reducing the concentration of a reagent is not allowed under this procedure because the impact of such a change on the chemistry of the procedure must be assessed prior to implementation.

Reduce the toxicity of waste by purchasing lower concentration stock standards, lower concentration stock reagents, and solutions to replace neat chemicals whenever possible. However, does not change the concentrations of standards and reagents specifically designated in this SOP.

4.4 Waste Management

The EPA Region 9 Laboratory complies with all applicable rules and regulations in the management of laboratory waste. The laboratory minimizes and controls all releases from hoods and bench operations. All analysts must collect and manage laboratory waste in a manner consistent with EPA Region 9 Laboratory SOP 706 *Laboratory Waste Management Procedure.* Solid and hazardous wastes are disposed of in compliance with hazardous waste identification rules and land disposal restrictions. If additional guidance is needed for new waste streams or changes to existing waste

streams, consult with EPA Laboratory Safety, Health, and Environmental Manager (LaSHEM) or ESAT Health and Safety and Environmental Compliance Task Manager or designees.

This procedure generates the following waste streams:

Waste Stream Description	Waste Label	Hazard Properties
Laboratory solid waste (gloves, contaminated paper towels, disposable glassware, etc.)	Non-hazardous Waste	Not applicable
SVOC Waste: Glass vials, dichloromethane, 0-2000 ppm semivolatile organic compounds	Hazardous Waste	Toxic

NOTE: N-Nitrosodimethylamine and 3,3'-dichlorobenzidine are listed as known or suspected carcinogens. As such, any sample or sample extract with a combined concentration of these compounds greater than or equal to 1000 mg/kg (wet weight) must be handled as an extremely hazardous waste. Should this concentration be determined, notify the LaSHEM (or designee).

5 SAMPLE HANDLING AND PRESERVATION

5.1 Containers and Required Sample Volume

Refer to EPA Region 9 Lab SOP 290 *Extraction of Soil Samples Using Pressurized Fluid* or SOP 265 *Extraction of Low Level Phenols and Polycyclic Aromatic Hydrocarbons from Water*.

5.2 Internal Chain-of-Custody

Sample extracts for GC/MS analysis are received from the extraction lab personnel and custody is transferred to the GC/MS laboratory staff. The GC/MS analyst acknowledges the receipt of the sample extracts by signing the appropriate sections of the completed LIMS bench sheet. Copies of tracking sheets, chain of custody records, and the original LIMS extraction bench sheet should accompany the sample extracts.

5.3 Sample Storage

Store extracts in the freezer in Room 402 or 406 at $\leq$ -10° C before and after analysis. Retain samples extracts until holding time has expired.

5.4 Holding Time

Extracts must be analyzed within 40 days of extraction.

6 INTERFERENCES

Method interferences can be caused by contaminants in solvents, reagents, glassware, and other sample processing apparatus. Phthalates are commonly found as laboratory contaminants. The analytical system must be demonstrated to be free from interferences under the conditions of the analysis by running a method blank (MB). The use of non-polytetrafluoroethylene (PTFE) tubing, non-PTFE thread sealants, or flow controllers with rubber components should be avoided.

Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed.

It is important that samples and standards be contained in the same solvent, i.e., the solvent for final working standards must be the same as the final solvent used in sample preparation. If this is not the case, chromatographic comparability of standards to samples may be affected.

7 APPARATUS AND MATERIALS

This section describes recommended apparatus and materials to be used for the analysis. All equipment, reagents, standards, and supplies must meet the technical and QC requirements of the reference method. Substitutions may be made provided that they are documented and equivalency is maintained.

7.1 Instruments and Equipment

7.1.1 GCMS System

7.1.1.1 Gas Chromatograph (GC): Agilent 6890, 6890N, 7890A, or equivalent. The GC must be capable of multilevel temperature programming and constant carrier gas flow throughout the temperature range. The GC should be equipped with an automatic sample injector, splitless injection port, and electronic pressure control (EPC).

7.1.1.2 GC column: 30 m, 0.25 mm ID, 0.5 µm DF. A fused silica capillary column with a bonded phase coating of 5% diphenyl 95% methyl silicone such as DB5, DB5MS, RTX 5, RXI-5Sil MS, HP 5, or equivalent. Any column capable of separating the target analytes and passing method QC without overloading at the concentration of the highest standard may be used.

7.1.1.3 Mass spectrometer: Agilent 5973, 5973N, 5975, or equivalent, capable of continuous scanning and selective ion monitoring from 35 to 500 amu every one second or less using 70 volts (nominal) electron energy in the electron impact ionization mode. The MS must be able to produce a mass spectrum that meets acceptance criteria when ≤50 ng of DFTPP is injected through the GC inlet.

7.1.1.4 Data system: ChemStation (available from Agilent), or equivalent, able to control the GC/MS system and to acquire, store, and reduce mass spectral data. The software must be able to process any GC/MS data file by recognizing a GC peak within a retention time window, comparing the mass spectrum from the GC peak with spectral data in a database. The software must also allow integration of the ion abundance of any specific ion between specified time or scan number limits and to calculate RRFs and concentrations of analytes in samples.

7.2 Reagents

Document the receipt of all reagents in the LIMS. A unique ID is assigned for each reagent. The reagent ID is reflected on all preparation and analysis batches.

Dichloromethane: High purity pesticide quality or equivalent. Recycled dichloromethane may be used if demonstrated to be suitable for use in this procedure.

7.3 Standards

Prepare standard solutions to include the project specific analytes of interest over the required concentration range. Document the receipt, preparation, and ampule open dates of all standards in the LIMS.

Store all ampulated calibration materials in the refrigerator at >0°C to ≤6°C protected from light. Use the manufacturer's expiration date for ampulated standards.

Store open stock ampules and working standards in a freezer at ≤-10 °C protected from light. The solution is assigned an expiration date which is either 6 months from preparation date, or the expiration date of the stock standard used in the preparation, whichever is earlier. Allow all standard solutions to equilibrate to room temperature before use.

7.3.1 Internal Standard Solution (IS): A solution of acenaphthene-d10, phenanthrene-d10, chrysene-d12, 1,4 dichlorobenzene-d4, perylene-d12, and naphthalene-d8 each in dichloromethane. Prepare by diluting purchased solutions to attain an extract concentration equivalent to the continuing calibration verification level. Restek 31206 or equivalent.

7.3.2 Surrogate solution: A solution of 2-fluorobiphenyl, nitrobenzene-d5, p-terphenyl-d14, and 1,2-dichlorobenzene-d4 (base/neutral surrogates); 2-fluorophenol, phenol-d5, 2,4,6-tribromophenol, and 2-chlorophenol-d4 (acid surrogates). Base/neutral surrogates - Restek 31072 or equivalent; acid surrogates - Restek 31073 or equivalent.

7.3.3 Calibration Stock Standard, Phenols: A solution of project-specific target analytes in dichloromethane.

- Restek 8270 Mega Mix Restek 31850,
- 4-Chlorophenol AccuStandard AS-E0183,
- 3,5-dichlorophenol Supelco 44-2378,
- 3,4-dichlorophenol Supelco 44-2375,
- 3,4,5-Trichlorophenol Accustandard M-1653-IS.

7.3.4 Calibration Stock Standard PAH: A solution of project-specific target analytes in dichloromethane. Restek 8270 Mega Mix Restek 31850.

7.3.5 Calibration standards: A solution of target analytes listed in Appendix B prepared by diluting the calibration stock standard and mixing it with the appropriate amount of IS solution to attain the project specific concentration range. The suggested target analyte concentrations are listed in the following table:

Analysis Type	Calibration Levels (ng/L)					
	1 (QLS)	2	3 (CCV)	4	5	6
PAH	50	100	500	1,000	5,000	8,000
Phenols	1,000	2,000	5,000	10,000	15,000	20,000

7.3.6 Second Source Standard (SCV): A solution of target analytes listed in Appendix B and surrogates prepared from a source different from the calibration standard. The SCV is used to check the accuracy of the initial calibration solutions.

- PAH: Supelco 47543-U, EPA 8310 Polynuclear Aromatic Hydrocarbons Mix, 2000 µg/mL) or equivalent.
- Phenols: Accustandard ACID MIX (CLP-HC-A-R, 2000 µg/mL) or equivalent.

Note: Some phenolic compounds are extremely difficult to obtain. Consequently, not every target analyte will necessarily be in the phenol SCV. For the phenol analysis at least one of each type of phenolic compound will be represented in the SCV (i.e., one di-chlorophenol, one tri-chlorophenol etc.). The PAH SCV will have every compound present.

7.3.7 GC/MS Tuning Solution (MS tune). A solution of DFTPP, 4,4'-DDT, pentachlorophenol, and benzidine at ≤50 ng/µL each in dichloromethane. Restek 31615 or equivalent.

7.4 Supplies

7.4.1 Syringes: 10 µL, 25 µL, 50 µL, 100 µL, 250 µL, 500 µL, 1 mL.

7.4.2 Helium carrier gas: Ultra-high purity, 99.999%.

8 ANALYTICAL PROCEDURES

8.1 Instrument Operation

Check the mass spectrometer for leaks on a daily basis, prior to the analysis of the tuning compound. Refer to Section 8.4 and Appendix F for system maintenance requirements.

Set-up the GC/MS following operating instructions provided by the manufacturer. Use the method provided in Appendix D as a starting point.

8.1.1 Mass axis calibration

Calibrate the Mass Axis of the MS prior to analyzing the DFTPP standard each day that samples are analyzed. Use the settings in the most recent tune file as the initial conditions; save the tune file using the naming convention in Appendix E and generate a tune report.

Since DFTPP is analyzed in the full scan mode, the most recent BNA method will be used for the analysis of DFTPP. Select the most recent BNA method and associate the newly generated tune file with this method; name the method using the naming convention in Appendix E and save it. Use this method for all subsequent DFTPP analyses.

Select the most recent low level SIM method (phenols or PAH, depending upon the procedure used) and associate the newly generated tune file with this method; name the method using the naming convention in Appendix E and save it. Use this method for all subsequent SIM analyses.

Refer to Section 9.2.1 and Appendix C for frequency, acceptance criteria, and corrective action requirements.

8.1.2 GC/MS tuning

The GC/MS system must meet the mass spectral ion abundance criteria for DFTPP prior to analysis. Proper tuning of the instrument is necessary to produce standardized fragmentation patterns of target compounds.

Inject 1 µL of the DFTPP solution using the BNA method provided in Appendix D.

Quantitate the DFTPP solution data and generate a DFTPP report using the Autofind DFTPP to printer menu item from the Tuner → Evaluate DFTPP menu.

The autofind procedure will automatically find the DFTPP peak, average three scans (the peak apex scan and the scans immediately preceding and following the apex), perform a background subtraction and print out a hard copy of the spectrum, the chromatogram, and the table of ion abundances.

Generate a tailing factor report for the pentachlorophenol and benzidine peaks.

Refer to Section 9.2.2 and Appendix C for frequency, acceptance criteria, and corrective action requirements.

8.2 Calibration and Standardization

8.2.1 Initial Calibration

Prior to analyzing an initial calibration, ensure that proper system maintenance and GC/MS tuning (DFTPPand/or manual tune) has been performed.

Since retention times may drift due to column maintenance or condition, it is necessary to verify the retention times for all compounds. To verify retention times, analyze a calibration standard in the full scan mode using the BNA method used for DFTPP analysis prior to analyzing the initial calibration. Adjust SIM groups' retention time windows accordingly.

When the instrument is ready for analysis, perform the following steps:

1. In the ChemStation data analysis module, load the current initial calibration method from D:\MSDCHEM\Year\Methods\
2. Perform an initial calibration using the recommended concentrations listed in Section 7.3.5.
3. Update the response factors in the method using the newly acquired calibration files.
4. Update the retention time in the method using the newly acquired continuing calibration level.
5. Update the qualifier ion relative responses from the continuing calibration level.

6. Save the method as outlined in the "ChemStation File Naming Convention"
7. Generate "Response Factor Report."
8. Check the calibration files listed on the "Response Factor Report" to insure that the correct files are being used.
9. Check the time and date to ensure that the correct update is used.
10. Process the SCV with the newly created initial calibration, check to make sure the "QLast Update" time and date match the "Response Factor Report"
11. Open and immediately close Chemsation Custom Reports. (note: no Chemstation custom reports are actually used, opening and closing custom reports populates "detail.xls" which is later mined for data.)
12. Open the latest copy of C:\msdchem\custrpt\ "Full_Custom_Report.xls" Make sure automatic updates of links is enabled under options.
13. Go to the "SCV Recovery","ICAL Area", "ICALconc", tabs and print reports. Make sure the SCV passes acceptance criteria (appendix C) and the ICAL areas match what is on the Chemstation quant reports.
14. Verify that the method was updated correctly. Print the Compound List Report from ChemStation. Verify that the average response factor is used. Scrupulously check the elution order and retention times, compare them to an old ICAL if needed.
15. Copy the method to I:\RoomNumber\Instrument\Year\Methods\
16. Manually calculate a result for one surrogate in the SCV to insure that the correct RFs are being used and write the results on the quantitation report.
17. Save a hard copy of the initial calibration files so they may be copied and included in associated packages.

Refer to Section 9.2.3 and Appendix C for frequency, acceptance criteria, and corrective action requirements.

8.2.2 Continuing Calibration Verification

Analyze a calibration verification standard at the beginning of each analytical sequence by performing the following steps:

1. In the ChemStation data analysis module, load today's method from D:\MSDCHEM\Year\Methods\
2. Acquire the continuing calibration using today's method.
3. Quantitate the continuing calibration verification file.
4. Generate "Evaluate Continuing Calibration Report".
5. Manually calculate a result for one surrogate to insure that the correct RFs are being used and write the results on the quantitation report.
6. As each run is quantitated during the day, make sure that the same date and time stamp (example: "QLast Update : Mon Jul 25 08:15:58 2011") is reflected on each file header.

7. If QLast Update time stamp changes, state the reason, repeat steps 4-6, and include the reports generated in the package.

Save a copy of the method to the LAN, when the data is backed up to the LAN the following day.

Refer to Section 9.2.4 and Appendix C for frequency, acceptance criteria, and corrective action requirements.

8.2.3 Quantitation Limit Verification Standard (CRL)

Analyze a quantitation limit standard at the concentration of the lowest point of the initial calibration for each analytical sequence.

Refer to Section 9.2.5 and Appendix C for frequency, acceptance criteria, and corrective action requirements.

8.3 Analysis

8.3.1 Sample Preparation

Allow the sample extracts to reach ambient temperature before analysis.

Check that the numbers on the vials coincide with the numbers on the LIMS extraction batch to ensure that the correct sample is being analyzed.

Note if the sample has an unusual color or other physical properties. If any physical signs of contamination are present, screen the samples to protect the analytical system from damage or contamination, and to determine the appropriate subsequent dilutions. Record unusual items in the LIMS work order memo .

Add appropriate amount of IS solutions to each field and QC sample extract to attain an IS extract concentration equivalent to the continuing calibration verification level.

8.3.2 Sample Analysis and Analytical Sequence

Obtain a LIMS sequence number by generating an empty LIMS sequence; specify the analysis and instrument, number associated with the samples to be analyzed.

Enter sample sequence in the instrument software. Include the laboratory sample number (work order-sample number) in the "Sample" field and dilution level, if any in the "Multiplier" field. Use the LIMS sequence number to name instrument QC as outlined in Appendix D.

Enter the BNA method name in the "Method" field; analyze the DFTPP sample with this method.

Enter the daily instrument SIM method name in the "Method" field; analyze all standards and samples with this method.

Name the data files according to the data file naming convention outlined in Appendix E.

Load the samples in the autosampler according to their designated positions in the sequence file. The recommended analysis sequence is:

1. DFTPP
2. CCV
3. QLS
4. Samples, sample dilutions, and or QC samples as needed
5. Instrument Blanks, as needed

8.3.3 Analyte Identification and Quantitation

8.3.3.1 Analyte Identification

In order for a target compound to be identified as present in a sample both the retention time and the characteristic ions of the peak must match those of the standard.

If a compound cannot be verified using these criteria but in the technical judgment of the analyst is present, report the analyte and include supporting evidence in the raw data package.

Cross out all reported hits that do not meet qualitative criteria and document the reason on the quantitation report.

Review the chromatogram for possible false negatives and edit results as needed.

8.3.3.2 Analyte Quantitation

Quantitate the data and print out ChemStation detailed quantitation reports and chromatograms. Use the average relative response factor from the initial calibration for quantitation.

Analyte concentrations in the sample extracts as shown on the ChemStation quantitation report are calculated as follow:

LIMS calculates final analyte concentrations in samples. To verify the LIMS reported values for water samples, calculate results for target analytes using the following equation:

$$\text{Conc. } (\mu g/L) = Ax * AMTIS * DF * 1000) / (AIS * RRF_{avg} * V)$$

Where:

Ax = area of the characteristic ion of the compound
AMTIS = amount of internal standard in µg/L
DF = dilution factor
AIS = area of the characteristic ion of the associated internal standard
RRF_{avg} = analyte average relative response factor from the initial calibration
V = Volume extracted in mL

For soil samples, calculate results for target analytes

$$\text{Conc.ug / Kg (dry weight basis)} = \frac{A_x \times C_{is} \times V_t \times V_i \times DF \times GPC}{A_{is} \times \overline{RRF} \times W \times D}$$

Where:

A_X = area of the quantitation ion of the compound
C_{IS} = concentration of Internal Standard in µg/L (500 µg/L)
D = dry weight factor (Percent solids/100)
W = weight of sample in grams
A_{IS} = area of the characteristic ion of the associated internal standard
RRF = analyte mean relative response factor from the initial calibration
Vt = volume of concentrated extract in µL
DF = dilution factor
GPC = GPC factor, normally 1.0 if not used, 2.0 if used
Vi = volume of extract injected in µL

8.3.3.3 Manual Integration

Where the chromatography software integrates the signal inconsistently, follow SOP 835, *Chromatographic Integration Procedures*. All manual chromatographic integration must be initialed and dated by the analyst and approved by the supervisor, Chemistry Technical Director, Quality Assurance Officer, or designees.

8.3.4 Data and QC Review

- Process and review the results for the CCV and CRL instrument QC samples. Print a ChemStation Evaluate Continuing Calibration Report using the appropriate settings to verify that the CCV results are within QC limits. Use Chemstation, Excel or LIMS to verify that the CRL is within QC limits. See Section9.2 for instrument QC requirements and Appendix C for acceptance criteria.

- Process and review the results for the MB, LCS, and MS/MSD batch QC samples and verify that the results are within QC limits. See Section 9.3 for Batch QC requirements.

- Determine if surrogate recoveries for field and QC samples are within QC limits. See Section 9.4 for Sample QC requirements.

- Review all sample results to determine if any samples need to be re-analyzed at a dilution.

- If a run is rejected for any reason, mark the raw data "Not Used" in large print and document the reason on the quantitation report.

- Qualify and flag results in the LIMS Data Entry/Review table following Appendix M of the Region 9 Quality Assurance Manual.

8.3.5 Data Export and LIMS Entry

- Generate epatemp.txt files for field and QC samples by printing the report to the screen; these files are used by the LIMS DataTool module to import the instrument results into the Data Entry/Review table.

- Copy sample data files from the local drive to the appropriate instrument data subdirectory on the Region 9 LAN to make them available to LIMS and to archive them.

- Populate the empty LIMS sequence file by editing the sequence using Data Tool to import the sequence information.

- Create an empty upload file containing the samples analyzed in the LIMS batch or sequence. Import and merge the data files using the LIMS DataTool module. Load the resulting merged data file into the LIMS Data Entry/Review table.

- Review results in the LIMS. Qualify and flag results in the LIMS Data Entry/Review table following Appendix M of the Region 9 Quality Assurance Manual.

8.4 Maintenance

The analyst should observe trends in the data such as declining response, erratic relative response, loss of classes of compounds, etc., which may signal the need for instrument maintenance. Document all routine maintenance or corrective actions taken in the maintenance logbook.

The following sections describe possible causes and corrective actions for common problems for GC and MS operations. Refer to Appendix F for routine preventative maintenance procedures and schedule.

8.4.1 GC Maintenance

Symptoms of common problems:

- Carryover
  Possible causes: Analyzing a sample containing high molecular weight components or analyzing high-level and low-level samples sequentially.
  Corrective action: As necessary, replace inlet liner, clean inlet, bake out inlet, bake out column, clip column, replace septum, replace column.

- Shorter retention time.
  Possible cause: column flow rate problem.
  Corrective action: check flow rate and adjust as necessary.

- Longer retention time and or smaller peaks.
  Possible causes: column flow rate problem, injection port leak, or column contamination.
  Corrective action: As necessary, check for leaks, replace septum, replace the liner, replace the lower injection port seal, and cut the column (a few inches to a foot or more) from the injector end. If issues remain, replace the column.

- Loss of resolution.
  Possible causes: column flow rate problem, injection port leak, or column contamination.
  Corrective action: Check for leaks, replace septum, liner, and inlet seal, clip the column (a few inches to a foot or more) from the injector end. If issues remain, replace the column.

8.4.2 MS maintenance:

Trend to be observed:

- Low m/z 502 to 69 ratio

- DFTPP ion 275 ratio is outside acceptance range
- Failing tune checks

Resolution: Clean the source.

9 QUALITY CONTROL

The EPA Region 9 Laboratory operates a formal quality control program and tracks compliance using the Lab QC Database. As it relates to this SOP, the QC program consists of a demonstration of capability, and the periodic analysis of MB, LCS, and other laboratory solutions as a continuing check on performance. The laboratory is required to maintain performance records that define the quality of the data that are generated. A summary of QC criteria is provided in Appendix C.

9.1 Demonstration of Capability

A Demonstration of Capability must be in place prior to using an analytical procedure and repeated if there is a change in instrument type, personnel, or method. Follow procedures described in EPA Region 9 Laboratory SOP 880.

9.2 Instrument QC

9.2.1 Mass calibration

Review the FC43 spectrum for compliance with the criteria list in Appendix C.

If the FC43 spectrum does not meet the criteria, corrective action must be taken. The corrective action may be as simple as adjusting the voltages/retuning the MS. If retuning the MS does not produce adequate FC43 spectra, further maintenance such as cleaning the ion source may be required.

9.2.2 GC/MS Tune

Review the DFTPP spectrum for compliance with the criteria list in Appendix C.

Locate the degradation products of 4,4'-DDT (4,4'-DDD and 4,4'-DDE. Calculate the breakdown of DDT using peak areas of each quantitation ion (Qion) in the following equation:

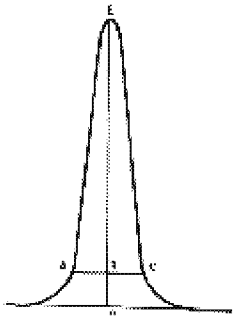
$$\%\ 4,4'\text{-DDT Breakdown} = \frac{\text{Total Qion Area (DDE + DDD)}}{\text{Total Qion Area (DDE + DDD + DDT)}} \times 100$$

where:

Qions of DDE: 246 Dalton, DDD: 235 Dalton, DDT: 235 Dalton

Benzidine and pentachlorophenol should be present at their normal responses. The tailing factor is calculated by the following equation:

$$TailingFactor = \frac{BC}{AB}$$



Where the peak is defined as follows: AC is the width at 10% height; DE is the height of peak and DB is the height at 10% of DE. This equation compares the width of the back half of the peak to the width of the front half of the peak at 10% of the height.

Refer to Appendix C for frequency, acceptance criteria, and corrective action requirements.

If the ion abundances, degradation, or tailing fail to meet the criteria, the DFTPP chromatogram should be examined for any obvious chromatographic problems (e.g., bad injection leading to poor response etc.). If the problem is related to poor chromatography, take the necessary corrective action and re-analyze the DFTPP. If the DFTPP continues to fail the ion abundance criteria, retune the mass spectrometer. It may also be necessary to clean the ion source or take other corrective action to achieve the ion abundance criteria.

If a sample is injected after the analytical time period has elapsed it must be re-analyzed.

9.2.3 Initial Calibration

Each GC/MS system must be calibrated whenever corrective action is performed which may change instrument response (e.g., ion source cleaning, column replacement, etc.) or if the continuing calibration acceptance criteria cannot be met.

Check the initial calibration for misidentified peaks due to retention time shifts. The most commonly misassigned pairs are benz(a)anthracene/chrysene and benzo(b)/benzo(k)fluoranthene.

No quantitation ion may saturate the detector.

The data system calculates the relative response factor (RRF) for each target compound and surrogate compound using the following equation:

$$RRF = (Ax)(Cis) / (Ais)(Cx)$$

Where

Ax = Area of quantitation ion of compound x. The recommended quantitation ions and internal standard assignments are listed in Appendix B.
Ais = Area of quantitation ion for associated internal standard
Cx = Concentration of compound x
Cis = Concentration of the associated internal standard

The data system calculates the average RRF (RRF_{avg}) for all analytes.

The data system calculates the percent relative standard deviation (%RSD) of the RRF values for each compound using the following equation.

$$\%RSD = (SD/ RRF_{avg})*100$$

Where

$$SD = \sqrt{\frac{\sum_{i=1}^{n}(x_i - x_{ave})^2}{n-1}}$$

As an exception, pentachlorophenol is calibrated using a quadratic curve. When this curve type is employed it is possible to report false positives on very low area counts. To prevent this error, analyze a standard at half of the quantitation limit along with the initial calibration. Note the area count for pentachlorophenol in this standard and do not report any results with less than this area count.

The %RSD, R^2, and SCV recovery requirements are listed in Appendix C.

If an ICAL fails because of one standard, a fresh solution of that standard may be analyzed and substituted for the standard that failed in the ICAL. If the failure is repeated (or the problem is not isolated to one calibration point), the system must be repaired so that the criteria are satisfied before any samples are analyzed.

If SCV criteria (see Appendix C) are not met, the SCV must be re-analyzed. If it fails again, prepare a fresh solution. If failure persists, take corrective action as needed, including reanalysis or re-preparation and reanalysis of the initial calibration if necessary.

9.2.4 Continuing Calibration Verification

Examine the areas of the quantitation ions of the internal standards in the calibration verification standard.

Refer to Appendix C for frequency, acceptance criteria, and corrective action requirements.

If the area for any internal standard does not meet the criteria, the CCV may be re-analyzed. If the failure is repeated, the analysis shall be terminated, the problem corrected, and a new calibration curve prepared.

Examine the retention times of internal standards in the calibration verification standard. If the retention time for any internal standard does not meet the criteria, inspect the chromatographic system for malfunctions and take corrective action as needed and prepare a new calibration curve.

The data system calculates the percent difference (%D) of the RRF values for each compound using the following equation:

$$\%D = \frac{RRF_c - RRF_{avg}}{RRF_{avg}} x\ 100$$

Where:

RRF_c = Relative Response Factor of compound c.
RRF_{avg} = Average Relative Response Factor.

If the continuing calibration does not meet %D criterion listed in Appendix C, the analysis shall be terminated, the problem corrected, and a new continuing calibration analyzed.

Qualify and flag results as needed in the LIMS Data Entry/Review table following Appendix M of the Region 9 Quality Assurance Manual.

9.2.5 Quantitation Limit Standard

QLS must be analyzed at the beginning of the analytical run, typically just after the CCV. The QLS concentrations match the QL concentration (at the instrument). The recovery of analytes in the QLS is calculated as:

$$\%R = \frac{M}{T} \times 100$$

Where

%R	=	percent recovery of the standard.
M	=	measured concentration of the analyte, ug/L.
T	=	true concentration of the analyte in the ug/L.

Check that the recoveries meet the criteria specified in Appendix C.

If the QLS recovery does not meet criteria provided in Appendix C, rerun the QLS once to verify. If still unacceptable determine the cause, take corrective action.

Qualify and flag results as needed in the LIMS Data Entry/Review table following Appendix M of the Region 9 Quality Assurance Manual.

9.3 Batch QC

9.3.1 Method Blank

- Extract and analyze a method blank (MB) with each extraction batch to demonstrate that the entire analytical system, from extraction through GC/MS analysis, is free of contamination.

- If the surrogate recovery exceeds acceptance criteria (high bias) and is ND, narrate the error and continue. If the surrogate recovery fails acceptance criteria low, re-analyze the MB. If the surrogate recovery still does not meet acceptance criteria, evaluate sample surrogate recoveries to determine acceptability. If samples have acceptable recovery and are ND, report. Otherwise, re-extract any sample that is ND and fails surrogate low or any sample that has a hit. Note that if any samples in the batch are reported, the MB must also be reported and a note placed in the LIMS work order memo field.

- Evaluate the MB as soon as possible after it has been analyzed to determine if the results are within QC limits. See Appendix C for QC limits.

- Corrective action - If the MB result exceeds QC limits, check the associated samples as follow:

    1. If the sample result is less than five times the MB result, re-analyze the MB. If the MB result still exceeds QC limits, the batch may have to be re-extracted. Consult with the Technical Director or designee.

2. If the sample result is greater than five times the MB result or is not detected, report the sample result.

9.3.2 Laboratory Control Sample

- Analyze a laboratory control sample (LCS) to demonstrate that the analytical system is in control. The LCS is an MB spiked with matrix spiking solution.

- Calculate the percent recovery (%R) using the following equation:

$$\%\ R = [MR/SA] \times 100$$

Where,
MR = Measured result
SA = Spike added

- The %R must be within the QC limits in Appendix C. If acceptable recoveries cannot be achieved, re-analyze the LCS. If the LCS result still exceeds QC limits, re-extract the LCS and all associated samples.

9.3.3 Matrix Spike/Matrix Spike Duplicate

- Matrix spike (MS) and matrix spike duplicate (MSD) samples are extracted and analyzed for each batch of twenty or fewer samples extracted as a group.

- Calculate the recovery of each analyte.

$$\%\ R = [(SSR - SR)/SA] \times 100$$

Where,
SSR = Spiked sample result
SR = Unspiked sample result
SA = Spike added

- Calculate the relative percent differences (RPD) of the recoveries of each analyte in the MS and MSD using the following equation:

$$RPD = \frac{(MSC - MSDC)}{(MSC + MSDC)/2} \times 100$$

Where,
MSC = Measured concentration of analyte in MS
MSDC = Measured concentration of analyte in MSD

- See Appendix C for QC limits.

  The MS/MSD recovery limits are advisory limits only. If the limits are not met, then no further action is required, as long as the LCS is within limits, since the purpose of these analyses is to determine matrix effects on compound recovery. However, frequent failure to meet the recovery or RPD criteria should alert the analyst that a problem may exist and must be investigated. The analyst should analyze the matrix spike solution and check the recoveries of the spike compounds. A new solution should be prepared if the recoveries are not within 20% of expected.

- The table below lists the action to be taken based on the LCS and MS/MSD results.

QC ACCEPTANCE MATRIX + = PASS – = FAIL								
CASE	1	2	3	4	5	6	7	8
LCS - % REC	+	+	+	+	–	–	–	–
MS/MSD -% REC	+	–	+	–	+	–	+	–
MS/MSD – RPD	+	+	–	–	+	+	–	–

Case 1: Extraction batch acceptable.
Case 2: Extraction batch acceptable; matrix effect confirmed.
Cases 3 & 4: Extraction batch is unsatisfactory. Investigate MS/MSD problem and document findings in the LIMS memo field.
Cases 5, 6, 7, & 8: Extraction batch rejected. If additional sample volume is available, the batch should be re-extracted.

9.4 Sample QC

9.4.1 Surrogate Recovery

Note: in some instances, samples extracted under EPA Region 9 Lab SOP 275 *Extraction of Water Samples by Continuous Liquid-Liquid Extraction* may be analyzed by this procedure. In this event, the surrogate concentrations will exceed the calibration range and will not be reported.

- Calculate the surrogate recovery in all field and QC samples immediately after analysis using the following formula:

  $$\%R = (\text{Amount Found}/\text{Amount Spiked}) \times 100.$$

- Take the following steps if surrogate recovery is not within the limits listed in Appendix C:

    1. Ensure that there are no calculation errors, and check the system performance.
    2. Re-analyze the sample if a system performance problem or calculation error is not evident. The sample may be diluted for re-analysis if examination of the chromatogram so indicates.

- Do not reanalyze undiluted samples with surrogate recoveries outside the limits if the diluted analysis with acceptable surrogate recoveries is being submitted.

- Do not re-analyze the MS/MSD samples, even if surrogate recoveries are outside the limits.

- If the sample associated with the MS/MSD analyses does not meet the surrogate recovery criteria, it should be re-analyzed only if the matrix spike and duplicate surrogate recoveries are within the limits. If the sample and spikes show the same pattern (i.e., outside the limits), then the sample does not need re-analysis.

- If the surrogate recoveries of the re-analysis are within limits, then the problem was within the laboratory's control. Report the results from the re-analysis and submit the data from both analyses. The problem must be documented in the LIMS MMO field.

- If the re-analysis does not solve the problem and additional sample volume is available, the failing samples should be re-extracted unless the sample is ND with failing high recovery.

- If sample re-extraction is unfeasible, or surrogate recoveries of the re-extraction are also outside the QC limits, report the results from the first analysis and submit the data from both analyses. Distinguish between the original analysis and the re-analysis by adding the "RE" suffix to the sample ID in the re-analysis. The problem must be documented in the LIMS MMO field.

9.4.2 Internal Standard Area:

- Evaluate the internal standard areas in all field and QC samples immediately after analysis.

- The internal standard areas must be within QC limits outlined in Appendix C.

- Take the following steps if the internal standard areas are not within the limits:

    1. Check the system performance.

2. Re-analyze the sample if a system performance problem is not evident. The sample may be diluted for re-analysis if examination of the chromatogram so indicates.

- Do not reanalyze undiluted samples with internal standard areas outside the limits if the diluted analysis with acceptable internal standard areas is being submitted.

- Do not re-analyze the MS/MSD samples, even if internal standard areas are outside the limits.

- If the sample associated with the MS/MSD analyses does not meet the internal standard areas criteria, it should be re-analyzed only if the matrix spike and duplicate internal standard areas are within the limits. If the sample and spikes show the same pattern (i.e., outside the limits), then the sample does not need re-analysis.

- If the internal standard areas of the re-analysis are within limits, then, the problem was within the laboratory's control. Report the results from the re-analysis and submit the data from both analyses. Distinguish between the analysis and re-analysis by adding an "RE" suffix to the sample ID on the re-analysis. The problem must be documented in the LIMS MMO field.

9.5 Method Performance

Refer to the table in Appendix G for a summary of method performance in the EPA Region 9 Laboratory from January 1, 2012 to December 31, 2013 for water samples. Insufficient data are available to calculate statistics for other matrices during this time period.

Functional areas of the SOP that may be significant sources of analytical error are:

1. Addition of internal standard: The amount and concentration of internal standard added is critical. The nominal concentration is used in calculating target analyte concentration.
2. Samples must be stored as outlined in the SOP to minimize analyte degradation and solvent evaporation.
3. Sample temperature: Sample extracts must be allowed to come up to room temperature prior to analysis. Failure to do so will cause heavy molecular weight analytes to precipitate thus reducing the observed concentration.
4. Poor column condition may results in inadequate analyte separation and inaccurate integration.

10 DOCUMENTATION

10.1 Standards

Record all standards (ICAL, ICV/CCV, QL, MS/MSD, and LCS) in the LIMS. A copy of each Analytical Standard Record associated with sample analysis must be available for printing in the data package.

10.2 Reagents

Record all reagents used for each analytical batch in the LIMS.

10.3 Analytical sequence

The analytical sequence is documented in the Element database or in the instrument Run Log, date of analysis, QC solution IDs, analyst initials, lab sample IDs, dilution factors and comments, if any, are recorded.

10.4 Analytical Report and Data Package

Analytical reports are produced using the LIMS. The data package is produced from LIMS and manual log records. EPA Region 9 Laboratory SOP 845 *Analytical Data Review* provides the typical format for data package deliverables.

10.5 Maintenance Logbook

Maintain a maintenance logbook for each instrument covered in this SOP. Document the following:

- Initial installation and performance
- Subsequent instrument modifications and upgrades, including major software upgrades.
- All preventative or routine maintenance performed including repairs and corrective or remedial actions. Whenever corrective action is taken, record the date, the problem and resolution, and documentation of return to control.

All entries should be made in accordance with EPA Region 9 Laboratory SOP 840, *Notebook Documentation and Control.*

10.6 SOP Distribution and Acknowledgement

After approval, make available an electronic copy of the final SOP to all laboratory staff expected to perform the SOP or review data generated by the SOP. (The Lab QC Database contains a list of assigned analysts for each SOP). All approved EPA

Region 9 Laboratory SOPs are maintained in the LotusNotes database in Adobe Acrobat portable document format.

Analyst training is documented via the Training Record form and the Read and Understood Signature log; the latter is entered into the Lab QC Database.

10.7 SOP Revisions

Revisions to this SOP are summarized in Appendix H.

11 REFERENCES

EPA Region 9 Laboratory documents (SOPs, the Laboratory Quality Assurance Plan, etc.) are not included in this list. Analysts are referred to the SOP database on LotusNotes or the local area network (G:\USER\SHARE\QA PROGRAM\LAB SOPS PDF) for these documents; laboratory users should contact the Chemistry Team Leader or Laboratory QAO for copies of any supporting documents.

Agilent 6890 Gas Chromatograph Users Manual

Agilent 7890 Gas Chromatograph Users Manual

Agilent 5973 MSD Hardware Manual

Agilent 5975 MSD Hardware Manual

Agilent Environmental Analysis User's Guide

Agilent EnviroQuant ChemStation User's Guide

USEPA Method 525.3, Determination of Semivolatile Organic Compounds in Drinking Water by Solid Phase Extraction and Capillary Column Gas Chromatography / Mass Spectrometry, Revision 1.0, February2012.

USEPA Method 3500C, Organic Extraction and Sample Preparation, Revision 3, February 2007.

USEPA Method 8000C, Determinative Chromatographic Separations, Revision 3, March 2003.

USEPA Method 8270D, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), Revision 4, February 2007.

APPENDIX A.
DEVIATIONS FROM THE REFERENCE METHOD

1. SW-846 method 3500C referenced by method 8270D specifies a concentration of 200 mg/L for the acid matrix spiking solution; this SOP specifies 100 mg/L.

2. Mass spectra used for target analytes are from the NIST library and are included in the reports to demonstrate that appropriate masses have been selected for quantitation and monitoring. However, the selected ion scans will not resemble the NIST spectra – comparison with the NIST spectra is not used for identification.

3. Relative Retention Time (RRT) of chromatographic peak is not evaluated in this SOP as documented in the reference method. ChemStation calculates a retention time window and does not calculate RRT window. The retention time window of ± 0.2 min in this procedure is more stringent than the ± 0.06 RRT units in the reference method.

APPENDIX B.
ANALYTES AND QUANTITATION LIMITS

The following is the target compounds list for semivolatile organics that have been detected using this SOP, as well as the internal standards and surrogates. Include are the CAS number, primary and secondary ion quantitation ions, associated internal standard and surrogate, and quantitation limits. The parameters below are recommended starting points and may be modified to meet method and project requirements. All parameters are documented in the ChemStation method.

Low Level PAH (polynuclear aromatic hydrocarbons):

Compound	CAS Number	Primary Quant Ion	Secondary Ion(s)	Int. Std	Surrogate	Water ng/L	Soil ug/kg	On Col. pg
Naphthalene	91-20-3	128	102	1	1	50	2.5	50
2-Methylnaphthalene	91-57-6	142	141	1	1	50	2.5	50
1-Methylnaphthalene	90-12-0	142	141	1	1	50	2.5	50
Acenaphthylene	208-96-8	152	151, 153	2	1	50	2.5	50
Acenaphthene	83-32-9	153	152, 154	2	1	50	2.5	50
Fluorene	86-73-7	166	165	2	1	50	2.5	50
Phenanthrene	85-01-8	178	179, 152	3	2	50	2.5	50
Anthracene	120-12-7	178	179, 152	3	2	50	2.5	50
Fluoranthene	206-44-0	202	101	3	2	50	2.5	50
Pyrene	129-00-0	202	101, 100	4	2	50	2.5	50
Benz(a)anthracene	56-55-3	228	229,114	4	2	50	2.5	50
Chrysene	218-01-9	228	114, 229	4	2	50	2.5	50
Benzo(b)fluoranthene	205-99-2	252	253, 126	5	2	50	2.5	50
Benzo(k)fluoranthene	207-08-9	252	253, 126	5	2	50	2.5	50
Benzo(a)pyrene	50-32-8	252	253, 126	5	2	50	2.5	50
Indeno(1,2,3-cd)pyrene	193-39-5	276	138	5	2	50	2.5	50
Dibenzo(a,h)anthracene	53-70-3	278	139, 279	5	2	50	2.5	50
Benzo(g,h,i)perylene	191-24-2	276	138, 277	5	2	50	2.5	50
Surrogates (as utilized)								
2-Fluorobiphenyl	321-60-8	172	171	2	1			
Terphenyl-d14	1718-51-0	244	122	4	2			

Compound	CAS Number	Primary Quant Ion	Secondary Ion(s)	Int. Std	Surrogate	Water ng/L	Soil ug/kg	On Col. pg
Internal Standards								
Naphthalene-d8	1146-65-2	136	108	1				
Acenaphthene-d10	15067-26-2	164	162, 160	2				
Phenanthrene-d10	1517-22-2	188	160	3				
Chrysene-d12	1719-03-5	240	120, 236	4				
Perylene-d12	1520-96-3	264	132, 265	5				

Low Level Phenols

Compound	CAS Number	Primary Quant Ion	Secondary Ion(s)	Int. Std	Surrogate	Water ug/L	Soil ug/kg	On Col. pg	Report
2-Chlorophenol	95-57-8	128	64,130	1	1	1	33	1000	X
3&4-Methylphenol	108-39-4/ 106-44-5	107	108,77	1	1	1	33	1000	X
2,4-Dichlorophenol	120-83-2	162	63,164	2	1	1	33	1000	X
3&4-Chlorophenol	108-43-0/ 106-48-9	128	65,130	2	1	1	33	1000	X
2,4,6-Trichlorophenol	88-06-2	196	198,97,132	3	2	1	33	1000	X
2,4,5-Trichlorophenol	95-95-4	196	198,97,132	3	2	1	33	1000	X
3,5-Dichlorophenol	591-35-5	162	164, 99,63	3	1	1	33	1000	X
3,4-Dichlorophenol	95-77-2	162	164, 99,63	3	1	1	33	1000	X
2,3,4,6-Tetrachlorophenol	58-90-2	232	230.131,133	3	2	1	33	1000	X
3,4,5-Trichlorophenol	609-19-8	196	198, 133,62	3	2	1	33	1000	X
Pentachlorophenol	87-86-5	265.9	264, 268	4	2	1	33	1000	X
Surrogates (as utilized)									
2-Chlorophenol-d4	93951-73-6	132	68,134	1	1				
2,4,6-Tribromophenol	118-79-6	330	332, 141,62	3	2				
Internal Standards									
1,4-Dichlorobenzene-d4	3855-82-1	150	152,115	1					
Naphthalene-d8	1146-65-2	136	108,54,68	2					
Acenaphthene-d10	15067-26-2	162	164, 160,80	3					
Phenanthrene-d10	1517-22-2	188	80, 94	4					

APPENDIX C.
QUALITY CONTROL PARAMETERS AND CRITERIA

<table>
<tr><th>ANALYSIS</th><th>SUMMARY</th><th>FREQUENCY</th></tr>
<tr><td>MS Tune (FC 43)</td><td><table><tr><th>Mass</th><th>Target % of Mass 69</th></tr><tr><td>50</td><td>0.3-5</td></tr><tr><td>69</td><td>100</td></tr><tr><td>131</td><td>20-120</td></tr><tr><td>219</td><td>20-120</td></tr><tr><td>414</td><td>0.3-10</td></tr><tr><td>502</td><td>0.3-10</td></tr></table></td><td>With every ICAL</td></tr>
<tr><td>GC/MS System Performance Check (DFTPP analysis)</td><td>The ion abundance ratios must meet the following criteria.<table><tr><th>Mass (m/z)</th><th>Relative Ion Abundance Criteria</th></tr><tr><td>51</td><td>10-80% of Base Peak</td></tr><tr><td>68</td><td>< 2% of mass 69</td></tr><tr><td>70</td><td>< 2% of mass 69</td></tr><tr><td>127</td><td>10-80% of Base Peak</td></tr><tr><td>197</td><td>< 2% of mass 198</td></tr><tr><td>198</td><td>Base peak, or > 50% of Mass 442</td></tr><tr><td>199</td><td>5-9% of mass 198</td></tr><tr><td>275</td><td>10-60% of Base Peak</td></tr><tr><td>365</td><td>> 1% of mass 198</td></tr><tr><td>441</td><td>present but < 24% of mass 442</td></tr><tr><td>442</td><td>Base Peak, or > 50% of mass 198</td></tr><tr><td>443</td><td>15-24% of mass 442</td></tr></table>Benzidine and pentachlorophenol responses should be at their expected levels in the DFTPP solution and the peak-tailing factor should be less than 2.
Breakdown criteria for DDT is less than 20%.</td><td>At the start of 12-hour analytical sequence</td></tr>
<tr><td>Initial Calibration</td><td>90% of reported analytes should meet the maximum %RSD of 20. If one or more analytes exceed the RSD limit, the initial calibration may still be acceptable if the following conditions are met:
The %RSD of the reported analytes that exceed the limit is ≤30
The mean of the %RSD values for all reported analytes is less than or equal to 20%.
Pentachlorophenol uses a quadratic curve fit. The coefficient of determination, R^2 must be ≥ 0.99</td><td>As needed</td></tr>
</table>

ANALYSIS	SUMMARY	FREQUENCY
Second Source Calibration Verification	Recovery for 90% of reported analytes must be within 70-130%. Data must be flagged as failing initial calibration criteria for analytes not within the acceptance limits.	Once with every ICAL
Continuing Instrument Calibration Verification	All reported analytes should meet a maximum %D of 20 and minimum RRF of 0.010. If one or more reported analytes exceed maximum %D limit, the calibration verification may still be acceptable if the following conditions are met: 90% of reported analytes meet a maximum %D of 20 The %D of the reported analytes that exceed the limit is ≤ 30 The mean of the %D values for all reported analytes is less than or equal to 20. Internal standard retention time should be within 30 seconds from that in the mid-point standard level of the most recent initial calibration. EICP area for any of the internal standards should be within (-50% to +100%) from that in the mid-point standard level of the most recent initial calibration sequence.	Once every 12-hours
Quantitation Limit Verification	The percent recovery for 90% of the reported analytes should be between 60 to 140 percent of the actual concentration.	Once every 12-hours
Method Blank (MB)/Instrument Blank	The MB is acceptable if it contains less than one-half the quantitation limit (QL) of all target compounds.	One per extraction batch
Target Analytes	All ions present in the standard or reference mass spectrum at a relative intensity of 20 % of the most abundant ion should be present in the sample spectrum if that ion is being monitored. The relative intensities of the ions in the sample mass spectrum should agree within 30% of the relative intensities of those ions that are monitored in the standard mass spectrum. For example, an ion with an abundance of 50% in the standard spectrum can have abundance between 20% and 80% in the sample spectrum.	
Internal Standards (covering reported analytes)	Compare the IS retention times and areas in the CCV standard to the mid-point standard of the most recent initial calibration. The retention time for any internal standard should be within 0.5 minute and the total area of internal standard should be recovered within -50% to +100% from the mid-point standard of the most recent	

ANALYSIS	SUMMARY	FREQUENCY
	initial calibration. Compare the IS retention times and areas in the field and QC samples analyzed within the 12-hour analytical period to the associated 12-hour CCV standard. The retention time for any internal standard should be within 0.5 minute and the total area of internal standard should be recovered within -50% to +100% from the associated 12-hour CCV standard.	
Laboratory control sample (LCS)	90% of reported analytes should meet the acceptance criteria or the batch may require re-extraction. Refer to the tables below for control limits.	One per batch or every 20 samples, whichever is more frequent.
Matrix Spike/ Matrix Spike Duplicate MS/MSD	Refer to the tables below for control limits. Flag outliers.	One per batch or every 20 samples, whichever is more frequent

LCS and MS/MSD QC Criteria:

LCS and MS/MSD criteria are based on results obtained from January 1, 2012 to December 31, 2013 for water samples. A default value of 20% will be used for the RPD for waters and 30% for other matrices. These limits will be applied to all matrices until sufficient data are available to calculate statistical limits.

Low level Polynuclear Aromatic Hydrocarbons

	LCS		Matrix Spike		
Analyte	**Lower**	**Upper**	**Lower**	**Upper**	**RPD**
1-Methylnaphthalene	20	110	37	110	20/30
2-Methylnaphthalene	20	110	40	110	20/30
Acenaphthene	25	110	45	111	20/30
Acenaphthylene	35	110	56	110	20/30
Anthracene	56	110	64	110	20/30
Benz(a)anthracene	70	125	42	136	20/30
Benzo(a)pyrene	62	110	32	115	20/30
Benzo(b)fluoranthene	63	111	28	117	20/30
Benzo(g,h,i)perylene	59	110	27	110	20/30
Benzo(k)fluoranthene	63	116	31	117	20/30
Chrysene	64	110	37	115	20/30
Dibenz(a,h)anthracene	60	121	32	117	20/30
Fluoranthene	65	110	43	123	20/30
Fluorene	36	110	51	112	20/30
Indeno(1,2,3-cd)pyrene	60	116	29	116	20/30
Naphthalene	20	110	25	116	20/30
Phenanthrene	49	110	55	110	20/30
Pyrene	57	114	34	134	20/30

Surrogate Recovery

Surrogate criteria are based on sample results obtained from January 1, 2012 to December 31, 2013 for water samples.

Surrogate	**Lower Percent**	**Upper Percent**
2-Fluorobiphenyl	32	110
Terphenyl-d14	39	136

Low level Phenols

	LCS		Matrix Spike		
Analyte	**Lower**	**Upper**	**Lower**	**Upper**	**RPD**
2,3,4,6-Tetrachlorophenol	49	110	52	132	20/30
2,4,5-Trichlorophenol	52	110	49	142	20/30
2,4,6-Trichlorophenol	51	110	33	134	20/30
2,4-Dichlorophenol	61	110	40	132	20/30
2-Chlorophenol	60	110	53	110	20/30
3&4-Chlorophenol	57	110	39	135	20/30
3&4-Methylphenol	63	110	20	148	20/30
3,4,5-Trichlorophenol	55	110	67	150	20/30
3,4-Dichlorophenol	55	110	55	136	20/30
3,5-Dichlorophenol	55	110	58	133	20/30
Pentachlorophenol	29	110	55	150	20/30

*Insufficient historical data to establish limit. Default values appear in the table.

Surrogate Recovery

Surrogate criteria are based on sample results obtained from January 1, 2012 to December 31, 2013 for water samples.

Surrogate	**Lower Percent**	**Upper Percent**
2,4,6-Tribromophenol	20	150
2-Chlorophenol-d4	26	125

APPENDIX D.
RECOMMENDED INSTRUMENT PARAMETERS

A. DFTPP Method

INSTRUMENT CONTROL PARAMETERS

6890 GC METHOD

OVEN
- Initial temp: 40 'C (On)
- Initial time: 2.00 min
- Maximum temp: 350 'C
- Equilibration time: 0.50 min
- Ramps:

#	Rate	Final temp	Final time
1	15.00	330	6.00
2	0.0 (Off)		

- Post temp: 0 'C
- Post time: 0.00 min
- Run time: 27.33 min

FRONT INLET (SPLIT/SPLITLESS)
- Mode: Pulsed Splitless
- Initial temp: 280 'C (On)
- Pressure: 6.03 psi (On)
- Pulse pressure: 30.0 psi
- Pulse time: 0.50 min
- Purge flow: 20.0 mL/min
- Purge time: 0.40 min
- Total flow: 23.5 mL/min
- Gas saver: On
- Saver flow: 20.0 mL/min
- Saver time: 2.00 min
- Gas type: Helium

BACK INLET (UNKNOWN)

COLUMN 1
- Capillary Column
- Model Number: Agilent 122-5536
- DB-5ms, 0.25mm * 30m * 0.5um
- Max temperature: 350 'C
- Nominal length: 30.0 m
- Nominal diameter: 250.00 um
- Nominal film thickness: 0.50 um
- Mode: constant flow
- Initial flow: 0.9 mL/min
- Nominal init pressure: 6.04 psi
- Average velocity: 34 cm/sec
- Inlet: Front Inlet
- Outlet: MSD
- Outlet pressure: vacuum

COLUMN 2
- (not installed)

FRONT DETECTOR ()

BACK DETECTOR ()

```
SIGNAL 1
   Data rate:  20 Hz
   Type:  test plot
   Save Data:  Off
   Zero:  0.0 (Off)
   Range:  0
   Fast Peaks:  Off
   Attenuation:  0

SIGNAL 2
   Data rate:  20 Hz
   Type:  test plot
   Save Data:  Off
   Zero:  0.0 (Off)
   Range:  0
   Fast Peaks:  Off
   Attenuation:  0

COLUMN COMP 1
   (No Detectors Installed)

COLUMN COMP 2
   (No Detectors Installed)

THERMAL AUX 2
   Use:  MSD Transfer Line Heater
   Description:
   Initial temp:  280 'C (On)
   Initial time:  30.00 min
      #  Rate  Final temp  Final time
      1   0.0(Off)

POST RUN
   Post Time: 0.00 min

TIME TABLE
   Time       Specifier                    Parameter & Setpoint

                              7673 Injector


     Front Injector:
        Sample Washes                0
        Sample Pumps                 3
        Injection Volume           1.0 microliters
        Syringe Size              10.0 microliters
        Nanoliter Adapter          Off
        PostInj Solvent A Washes     3
        PostInj Solvent B Washes     3
        Viscosity Delay              0 seconds
        Plunger Speed             Fast
        PreInjection Dwell        0.00 minutes
        PostInjection Dwell       0.00 minutes

     Back Injector:
No parameters specified

 Column 1 Inventory Number :
 Column 2 Inventory Number :

                              MS ACQUISITION PARAMETERS


General Information
------- -----------

Tune File                : 040214DFT.U
Acquistion Mode          : Scan
```

```
MS Information
-- -----------

Solvent Delay            : 3.00 min

EM Absolute              : True
Resulting EM Voltage     : 1200.0

[Scan Parameters]

Low Mass                 : 50.0
High Mass                : 550.0
Threshold                : 500
Sample #                 : 2       A/D Samples    4
Plot 1 low mass          : 50.0
Plot 1 high mass         : 550.0

[MSZones]

MS Quad                  : 200 C   maximum 200 C
MS Source                : 250 C   maximum 250 C

                        END OF MS ACQUISITION PARAMETERS


                          TUNE PARAMETERS
                       ----------------------------

 EMISSION     :      34.610
 ENERGY       :      69.922
 REPELLER     :      28.783
 IONFOCUS     :      90.157
 ENTRANCE_LE  :       0.000
 EMVOLTS      :    1141.176
 AMUGAIN      :    2150.000
 AMUOFFSET    :     126.000
 FILAMENT     :       1.000
 DCPOLARITY   :       0.000
 ENTLENSOFFS  :    20.078@ 50    13.553@ 69    14.055@131    12.549@219
10.290@414    10.039@502
MASSGAIN      :     241.000
MASSOFFSET    :     -15.000
```

B. PAH Method

INSTRUMENT CONTROL PARAMETERS

6890 GC METHOD

OVEN
- Initial temp: 40 'C (On)
- Initial time: 2.00 min
- Maximum temp: 350 'C
- Equilibration time: 0.50 min
- Ramps:

#	Rate	Final temp	Final time
1	25.00	150	0.00
2	15.00	330	6.00
3	0.0	(Off)	

- Post temp: 0 'C
- Post time: 0.00 min
- Run time: 24.40 min

FRONT INLET (SPLIT/SPLITLESS)
- Mode: Pulsed Splitless
- Initial temp: 280 'C (On)
- Pressure: 6.03 psi (On)
- Pulse pressure: 30.0 psi
- Pulse time: 0.50 min
- Purge flow: 20.0 mL/min
- Purge time: 0.40 min
- Total flow: 23.5 mL/min
- Gas saver: On
- Saver flow: 20.0 mL/min
- Saver time: 2.00 min
- Gas type: Helium

BACK INLET (UNKNOWN)

COLUMN 1
- Capillary Column
- Model Number: Agilent 122-5536
- DB-5ms, 0.25mm * 30m * 0.5um
- Max temperature: 350 'C
- Nominal length: 30.0 m
- Nominal diameter: 250.00 um
- Nominal film thickness: 0.50 um
- Mode: constant flow
- Initial flow: 0.9 mL/min
- Nominal init pressure: 6.04 psi
- Average velocity: 34 cm/sec

COLUMN 2
- (not installed)

```
   Inlet:  Front Inlet
   Outlet:  MSD
   Outlet pressure:  vacuum

FRONT DETECTOR ()                          BACK DETECTOR ()

SIGNAL 1                                   SIGNAL 2
   Data rate:  20 Hz                          Data rate:  20 Hz
   Type:  test plot                           Type:  test plot
   Save Data:  Off                            Save Data:  Off
   Zero:  0.0 (Off)                           Zero:  0.0 (Off)
   Range:  0                                  Range:  0
   Fast Peaks:  Off                           Fast Peaks:  Off
   Attenuation:  0                            Attenuation:  0

COLUMN COMP 1                              COLUMN COMP 2
   (No Detectors Installed)                   (No Detectors Installed)

THERMAL AUX 2
   Use:  MSD Transfer Line Heater
   Description:
   Initial temp:  280 'C (On)
   Initial time:  30.00 min
      #  Rate  Final temp  Final time
      1   0.0(Off)

                                           POST RUN
                                              Post Time: 0.00 min

TIME TABLE
   Time       Specifier                        Parameter & Setpoint


                              7673 Injector


     Front Injector:
        Sample Washes                 0
        Sample Pumps                  3
        Injection Volume            1.0 microliters
        Syringe Size               10.0 microliters
        Nanoliter Adapter           Off
        PostInj Solvent A Washes      3
        PostInj Solvent B Washes      3
        Viscosity Delay               0 seconds
        Plunger Speed              Fast
        PreInjection Dwell         0.00 minutes
        PostInjection Dwell        0.00 minutes

     Back Injector:
No parameters specified

 Column 1 Inventory Number :
 Column 2 Inventory Number :

                            MS ACQUISITION PARAMETERS
```

```
General Information
------- -----------

Tune File               : 041714DFT.U
Acquistion Mode         : SIM


MS Information
-- -----------

Solvent Delay           : 3.60 min

EM Absolute             : True
Resulting EM Voltage    : 1247.1

[Sim Parameters]

GROUP 1
Group ID                : 21
Resolution              : High
Plot 1 Ion              : 136.0
Ions/Dwell In Group     (  Mass,  Dwell) (  Mass,  Dwell) (  Mass,
Dwell)
                        ( 102.0,     25) ( 108.0,     25) ( 128.0,
25)
                        ( 136.0,     25) ( 141.0,     25) ( 142.0,
25)
                        ( 171.0,     25) ( 172.0,     25)

GROUP 2
Group ID                : 43
Resolution              : High
Group Start Time        : 10.16
Plot 1 Ion              : 151.0
Ions/Dwell In Group     (  Mass,  Dwell) (  Mass,  Dwell) (  Mass,
Dwell)
                        ( 139.0,     25) ( 151.0,     25) ( 152.0,
25)
                        ( 153.0,     25) ( 154.0,     25) ( 160.0,
25)
                        ( 162.0,     25) ( 164.0,     25) ( 168.0,
25)

GROUP 3
Group ID                : 52
Resolution              : High
Group Start Time        : 11.14
Plot 1 Ion              : 166.0
Ions/Dwell In Group     (  Mass,  Dwell) (  Mass,  Dwell) (  Mass,
Dwell)
                        ( 152.0,     25) ( 160.0,     25) ( 165.0,
25)
                        ( 166.0,     25) ( 167.0,     25) ( 178.0,
25)
```

```
                          ( 179.0,     25) ( 188.0,     25)

GROUP 4
Group ID                  : 66
Resolution                : High
Group Start Time          : 13.94
Plot 1 Ion                : 101.0
Ions/Dwell In Group       (  Mass,  Dwell) (  Mass,  Dwell) (  Mass,
Dwell)
                          ( 100.0,     25) ( 101.0,     25) ( 122.0,
25)
                          ( 202.0,     25) ( 244.0,     25)

GROUP 5
Group ID                  : 71
Resolution                : High
Group Start Time          : 16.58
Plot 1 Ion                : 240.0
Ions/Dwell In Group       (  Mass,  Dwell) (  Mass,  Dwell) (  Mass,
Dwell)
                          ( 114.0,     25) ( 120.0,     25) ( 228.0,
25)
                          ( 229.0,     25) ( 236.0,     25) ( 240.0,
25)

GROUP 6
Group ID                  : 77
Resolution                : High
Group Start Time          : 18.35
Plot 1 Ion                : 264.0
Ions/Dwell In Group       (  Mass,  Dwell) (  Mass,  Dwell) (  Mass,
Dwell)
                          ( 126.0,     25) ( 132.0,     25) ( 252.0,
25)
                          ( 253.0,     25) ( 264.0,     25) ( 265.0,
25)

GROUP 7
Group ID                  : 80
Resolution                : High
Group Start Time          : 20.61
Plot 1 Ion                : 278.0
Ions/Dwell In Group       (  Mass,  Dwell) (  Mass,  Dwell) (  Mass,
Dwell)
                          ( 138.0,     25) ( 139.0,     25) ( 276.0,
25)
                          ( 277.0,     25) ( 278.0,     25) ( 279.0,
25)

[MSZones]

MS Quad                   : 200 C   maximum 200 C
MS Source                 : 250 C   maximum 250 C


                          END OF MS ACQUISITION PARAMETERS
```

TUNE PARAMETERS

EMISSION	:	34.610
ENERGY	:	69.922
REPELLER	:	28.783
IONFOCUS	:	90.157
ENTRANCE_LE	:	0.000
EMVOLTS	:	1141.176
AMUGAIN	:	2150.000
AMUOFFSET	:	126.000
FILAMENT	:	1.000
DCPOLARITY	:	0.000
ENTLENSOFFS	:	20.078@ 50 13.553@ 69 14.055@131 12.549@219 10.290@414 10.039@502
MASSGAIN	:	229.000
MASSOFFSET	:	-15.000

END OF TUNE PARAMETERS

END OF INSTRUMENT CONTROL PARAMETERS

C. Phenol Method

```
                              INSTRUMENT CONTROL PARAMETERS:    AG5973L
                              ---------------------------------------

   C:\MSDCHEM\1\2014\METHOD\041814PCP_M&B.M
      Fri Apr 18 16:15:56 2014

Control Information
------- -----------

Sample Inlet      :  GC
Injection Source  :  GC ALS
Mass Spectrometer :  Enabled

================================================================================
===
                                    6890 GC METHOD
================================================================================
===


OVEN
   Initial temp:  40 'C (On)                Maximum temp:  350 'C
   Initial time:  0.00 min                  Equilibration time:  0.00 min
   Ramps:
      #  Rate  Final temp  Final time
      1 15.00      220        0.00
      2 25.00      330        6.00
      3   0.0(Off)
   Post temp:  50 'C
   Post time:  0.00 min
   Run time:  22.40 min


FRONT INLET (SPLIT/SPLITLESS)           BACK INLET (UNKNOWN)
   Mode:  Pulsed Splitless
   Initial temp:  275 'C (On)
   Pressure:  7.06 psi (On)
   Pulse pressure:  30.0 psi
   Pulse time:  0.50 min
   Purge flow:  40.0 mL/min
   Purge time:  0.40 min
   Total flow:  43.8 mL/min
   Gas saver:  Off
   Gas type:  Helium


COLUMN 1                                COLUMN 2
   Capillary Column                        (not installed)
   Model Number:  Restek RTX-1
   30m X 0.25MM X 0.25df
   Max temperature:  350 'C
```

```
   Nominal length:  30.0 m
   Nominal diameter:  250.00 um
   Nominal film thickness:  0.25 um
   Mode:  constant flow
   Initial flow:  1.0 mL/min
   Nominal init pressure:  7.07 psi
   Average velocity:  36 cm/sec
   Inlet:  Front Inlet
   Outlet:  MSD
   Outlet pressure:  vacuum


FRONT DETECTOR (NO DET)                    BACK DETECTOR (NO DET)


SIGNAL 1                                   SIGNAL 2
   Data rate:  20 Hz                          Data rate:  20 Hz
   Type:  test plot                           Type:  test plot
   Save Data:  Off                            Save Data:  Off
   Zero:  0.0 (Off)                           Zero:  0.0 (Off)
   Range:  0                                  Range:  0
   Fast Peaks:  Off                           Fast Peaks:  Off
   Attenuation:  0                            Attenuation:  0


COLUMN COMP 1                              COLUMN COMP 2
   (No Detectors Installed)                   (No Detectors Installed)


THERMAL AUX 2
   Use:  MSD Transfer Line Heater
   Description:
   Initial temp:  280 'C (On)
   Initial time:  0.00 min
      #  Rate  Final temp  Final time
      1   0.0(Off)


                                           POST RUN
                                              Post Time: 0.00 min


TIME TABLE
   Time       Specifier                        Parameter & Setpoint


                              GC Injector


     Front Injector:
        Sample Washes                 0
        Sample Pumps                  3
        Injection Volume           1.00 microliters
        Syringe Size               10.0 microliters
```

```
        PreInj Solvent A Washes       1
        PreInj Solvent B Washes       1
        PostInj Solvent A Washes      3
        PostInj Solvent B Washes      3
        Viscosity Delay               0 seconds
        Plunger Speed              Slow
        PreInjection Dwell         0.00 minutes
        PostInjection Dwell        0.00 minutes


     Back Injector:
No parameters specified

 Column 1 Inventory Number :
 Column 2 Inventory Number :

                                MS ACQUISITION PARAMETERS


General Information
------- -----------

Tune File                : 041814DFT.U
Acquistion Mode          : SIM


MS Information
-- -----------

Solvent Delay            : 4.30 min

EMV Mode                 : Absolute
Resulting EM Voltage     : 1200

[Sim Parameters]

GROUP 1
Group ID                 : 1
Resolution               : High
Plot 1 Ion               : 64.00
Ions/Dwell In Group      (  Mass,  Dwell) (  Mass,  Dwell) (  Mass,
Dwell)
                         ( 64.00,     25) ( 68.00,     25) ( 77.00,
25)
                         (107.00,     25) (108.00,     25) (115.00,
25)
                         (128.00,     25) (130.00,     25) (132.00,
25)
                         (134.00,     25) (150.00,     25) (152.00,
25)

GROUP 2
Group ID                 : 2
Resolution               : High
Group Start Time         : 7.36
Plot 1 Ion               : 54.00
```

```
Ions/Dwell In Group      (  Mass,  Dwell) (  Mass,  Dwell) (  Mass,
Dwell)
                         ( 54.00,     25) ( 63.00,     25) ( 65.00,
25)
                         ( 68.00,     25) ( 98.00,     25) (108.00,
25)
                         (128.00,     25) (130.00,     25) (136.00,
25)
                         (162.00,     25) (164.00,     25)

GROUP 3
Group ID                 : 3
Resolution               : High
Group Start Time         : 9.32
Plot 1 Ion               : 63.00
Ions/Dwell In Group      (  Mass,  Dwell) (  Mass,  Dwell) (  Mass,
Dwell)
                         ( 63.00,     25) ( 80.00,     25) ( 97.00,
25)
                         ( 99.00,     25) (132.00,     25) (160.00,
25)
                         (162.00,     25) (164.00,     25) (196.00,
25)
                         (198.00,     25)

GROUP 4
Group ID                 : 4
Resolution               : High
Group Start Time         : 11.63
Plot 1 Ion               : 332.00
Ions/Dwell In Group      (  Mass,  Dwell) (  Mass,  Dwell) (  Mass,
Dwell)
                         ( 62.00,     25) (131.00,     25) (133.00,
25)
                         (141.00,     25) (196.00,     25) (198.00,
25)
                         (230.00,     25) (232.00,     25) (330.00,
25)
                         (332.00,     25)

GROUP 5
Group ID                 : 5
Resolution               : High
Group Start Time         : 13.13
Plot 1 Ion               : 80.00
Ions/Dwell In Group      (  Mass,  Dwell) (  Mass,  Dwell) (  Mass,
Dwell)
                         ( 80.00,     25) ( 94.00,     25) (188.20,
25)
                         (264.00,     25) (265.90,    100) (268.00,
25)

[MSZones]

MS Source                : 230 C   maximum 250 C
MS Quad                  : 150 C   maximum 200 C
```

END OF MS ACQUISITION PARAMETERS

TUNE PARAMETERS for SN: US21863784

Trace Ion Detection is OFF.

```
EMISSION     :      34.610
ENERGY       :      69.922
REPELLER     :      25.767
IONFOCUS     :      78.769
ENTRANCE_LE  :       0.000
EMVOLTS      :    1200.000
                              Actual EMV  :    1200
                              GAIN FACTOR :       2.39
AMUGAIN      :    1944.000
AMUOFFSET    :     122.000
FILAMENT     :       1.000
DCPOLARITY   :       0.000
ENTLENSOFFS  :     13.804@  3    13.804@ 50    12.298@ 69    11.796@131
13.804@219    15.812@414    15.059@502    15.059@799
MASSGAIN     :    -153.000
MASSOFFSET   :      -8.000
```

END OF TUNE PARAMETERS

END OF INSTRUMENT CONTROL PARAMETERS

APPENDIX E.
CHEMSTATION FILE NAMING CONVENTIONS

Files for data, methods, tunes, and sequences on ChemStation computers and the LAN are named using the following naming conventions:

Directories

On the Workstation (When available, use D: drive):

Data:	C:\MSDCHEM \1\DATA\YEAR\DATA\MMDDYYSS or D:\MSDCHEM\YEAR\DATA\MMDDYYSS
Methods:	C:\MSDCHEM\1\DATA\YEAR\METHODS or D:\MSDCHEM\YEAR\METHODS
Sequences:	C:\MSDCHEM\1\DATA\YEAR\SEQUENCE or D:\MSDCHEM\YEAR\SEQUENCE
Tunes:	C:\MSDCHEM\1\5973N or C:\MSDCHEM\1\5975

On the LAN:

Data:	I:\DATA\ROOM NUMBER\INSTRUMENT\YEAR\DATA\MMDDYYSS
Methods:	I:\DATA\ROOM NUMBER\INSTRUMENT\YEAR\METHODS
Sequences:	I:\ DATA\ROOM NUMBER\INSTRUMENT\YEAR\SEQUENCE
Tunes:	I:\ DATA\ROOM NUMBER\INSTRUMENT\YEAR\TUNE

Methods
MMDDYYATC

Sequence
MMDDYYCSS

Data Files
MMDDYYCSS

Tune Files
MMDDYYA

Variables

A: Enter analysis, as follow:

504	EDB
TO15	TO15
BNA	BNA
BNA (SIM)	PAH or PCP
PEST	PEST
PCB	PCB
RSK175	RSK
TPH-G	GRO
TPH-D	DRO
VOA	VOA

BFB	BFB
DFTPP	DFT

C: Channel (use when applicable):

Front	A
Back	B
Both	AB

DD: Day i.e. 01, 02, 03, ….

MM: Month i.e. 01, 02, 03, ….

SS: Sequential number 01, 02, 03, ….

T: Matrix Type (if applicable)

Water	W
Solid	S
Air	A
Oil	O
Other	X

YY: Year i.e. 12 for 2012

APPENDIX F.
PREVENTATIVE MAINTENANCE REQUIREMENTS

GC Maintenance

Item	Frequency	Actions/Comments
Gas purifiers (carrier gas & detector gas)	Annually	Replacement schedule is based on capacity and grade of gases. In general, replace non-indicating traps every 6-12 months or when indicating traps start to change color. Replace indicating traps when indicating material is spent.
Split vent trap	Annually	Replace.
Syringes and/or syringe needles	As Needed	Replace syringe if dirt is noticeable in the syringe, if it cannot be cleaned, if the plunger doesn't slide easily, or if clogged. Replace needle if septa wear is abnormal or the needle becomes clogged.
Inlet liner	With each ICAL	Check often. Replace when dirt is visible in the liner or if chromatography is degraded.
Liner O-rings	With each ICAL	Replace with liner or with signs of wear.
Inlet septum	Daily (when analyzing samples)	Check often. Replace when signs of deterioration are visible (gaping holes, fragments in inlet liner, poor chromatography, low column pressure, etc.).
Inlet Hardware	Annually	Check for leaks and clean. Check parts and replace when parts are worn, scratched, or broken.
Column Maintenance	With each ICAL	Remove 1/2-1 meter from the front of the column when experiencing chromatographic problems (peak tailing, decreased sensitivity, retention time changes, etc.).
Solvent rinse	As needed	When chromatography degradation is due to column contamination. Only for bonded and cross-linked phases.
Replacement	As needed	When trimming and/or solvent rinsing no longer return chromatographic performance.
Ferrules		Replace ferrules when changing columns and inlet/detector parts.

MS Maintenance

Maintenance schedule

Task	Every week	Every 6 months	Every year	As needed
Tune the MSD				✓
Check the foreline pump oil level	✓			
Check the calibration vial(s)		✓		
Replace the foreline pump oil[1]		✓		
Clean the ion source				✓
Check the carrier gas trap(s) on the GC				✓
Replace the worn out parts				✓
Lubricate sideplate or vent valve O-rings[2]				✓

1 Every 3 months for CI MSDs using ammonia reagent gas.

2 Vacuum seals other than the side plate O-ring and vent valve O-ring do not need to be lubricated. Lubricating other seals can interfere with their correct function.

APPENDIX G.
METHOD PERFORMANCE

Low level Polynuclear Aromatic Hydrocarbons

Analyte	Number of Points	Mean	StdDev	Lower Performance	Upper Performance
1-Methylnaphthalene	20	49.1	10.9	27.2	70.9
2-Methylnaphthalene	35	48.5	11.4	25.7	71.3
Acenaphthene	35	57.8	10.9	36.1	79.5
Acenaphthylene	20	65.6	10.2	45.2	86.0
Anthracene	20	78.8	7.6	63.5	94.0
Benzo(a)anthracene	35	97.7	9.1	79.4	116.0
Benzo(a)pyrene	36	85.8	8.0	69.7	102.0
Benzo(b)fluoranthene	35	87.1	7.9	71.2	103.0
Benzo(g,h,i)perylene	35	84.4	8.6	67.3	102.0
Benzo(k)fluoranthene	35	89.8	8.8	72.2	107.0
Chrysene	35	85.7	7.2	71.3	100.0
Dibenz(a,h)anthracene	35	90.6	10.2	70.1	111.0
Fluoranthene	35	85.7	7.0	71.6	99.7
Fluorene	35	66.3	10.0	46.2	86.3
Indeno(1,2,3-cd)pyrene	35	88.2	9.4	69.4	107.0
Naphthalene	35	51.9	10.6	30.7	73.2
Phenanthrene	35	72.8	8.0	56.8	88.7
Pyrene	35	85.5	9.6	66.3	105.0

Low level Phenols

Analyte	Number of Points	Mean	StdDev	Lower Performance	Upper Performance
2,3,4,6-Tetrachlorophenol	18	73.3	8.1	57.1	89.5
2,4,5-Trichlorophenol	23	77.7	8.5	60.8	94.6
2,4,6-Trichlorophenol	23	76.7	8.5	59.6	93.7
2,4-Dichlorophenol	23	81.0	6.7	67.5	94.5
2-Chlorophenol	23	79.8	6.5	66.8	92.9
3&4-Chlorophenol	23	79.0	7.3	64.3	93.6
3&4-Methylphenol	23	85.4	7.5	70.4	100.0
3,4,5-Trichlorophenol	23	82.4	9.0	64.4	100.0
3,4-Dichlorophenol	23	78.7	8.0	62.8	94.7
3,5-Dichlorophenol	23	78.3	7.7	62.9	93.7
Pentachlorophenol	24	66.0	12.2	41.5	90.5

NOTE: The above data are from analysis of LCS in water for January 1, 2012 to December 31, 2013.

APPENDIX H.
REVISION HISTORY

STANDARD OPERATING PROCEDURE: 375
Revision: 5, Effective: 05/09/14

Low Level Semivolatile Organics Analysis

Revision	Effective Date	Description
3	04/13/09	Complete update of procedure to reflect multiple changes in the method. Included phenols, added method performance data, updated procedures for analytical methods.
4	02/01/12	1. Replaced Method Performance and QC Limits with recent data. 2. Updated tune criteria to Method 8270D recommendations. 3. Miscellaneous edits throughout for consistency with EPA Region 9 Laboratory practices (internal COC, waste labeling, etc.).
5	05/09/14	1. Updated Method Performance and QC limits and instrument parameters. 2. Changed DFTPP injection amount to prevent overloading. 3. Miscellaneous edits throughout for consistency with EPA Region 9 Laboratory practices (SOP 850, internal COC, waste labeling, etc.). 4. Phenol, 2-methylphenol, and 2,4-dimethylphenol were removed from the low level phenol method.